भारतीय मानक
Indian Standard

IS 4472 (Part 2) : 2021

वस्त्र रंजक सामग्री — वस्त्र सामग्रियों पर रंजकों के अनुप्रयोग वर्ग के पहचान की विधि

भाग 2 ऊन, रेशम एवं अन्य प्रोटीन रेशे

(पहला पुनरीक्षण)

Textile Dyestuffs — Identification of the Application Classes of Dyes on Textile Materials

Part 2 Wool, Silk and Other Protein Fibres

(First Revision)

ICS 59.040

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Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

FOREWORD

This Indian Standard (Part 2) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Textile Speciality Chemicals and Dyestuffs Sectional Committee had been approved by the Textiles Division Council.

This standard was first published in 1968 and the present revision has been taken up to update the method taking into consideration the technological developments since the publication.

This Indian Standard is published in three parts. The other parts in this series are:

Part 1 Cotton and other cellulosic fibres
Part 3 Man-made fibres

The methods of identification of dyes on wool, silk and other protein fibres prescribed in this standard are simple and are meant for rapid identification of application classes of dyes. Successful identification of application classes of dyes depends on experience and familiarity of the dyestuffs. The knowledge of a fibre eliminates dyes which are not applicable to the particular fibre and the identification is made somewhat easier.

The methods prescribed here do not involve the use of microscope, however, its use is not precluded if desired. In general, the identification of application classes of dyes is not dependent on any single test and final confirmation of the identity of an unknown class of dye should preferably be made by comparison with an authentic sample.

The composition of the Committee responsible for the formulation of this standard is given at Annex E.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

TEXTILE DYESTUFFS — IDENTIFICATION OF THE APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS

PART 2 WOOL, SILK AND OTHER PROTEIN FIBRES

(First Revision)

1 SCOPE

This standard (Part 2) prescribes methods for identification of application classes of dyestuff on wool, silk and other protein fibres.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
336 : 1973	Specification for ether (*second revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
1919 : 1982	Specification for sodium hydrosulphite (*first revision*)

3 PREPARATION OF TEST SPECIMEN

3.1 If the sample under test is fibre or yarn, take a tuft of fibre or yarn of about 3 cm in length.

3.2 If the sample under test is fabric, take a 3 × 3 cm test piece.

NOTES

1 In case of multi-coloured woven fabrics, the different coloured yarn should be identified separately.

2 In case of printed fabrics the sample should be taken from the printed portion of the sample.

3.3 In the case of finished textiles, the sample should be treated twice with 1 percent hydrochloric acid at boil for 5 min.

4 REAGENTS

4.0 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS 1070 : 1992) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

4.1 Acetic Acid — (a) 5 percent (*w/v*), and (b) glacial.

4.2 Ammonium Hydroxide Solution — Prepared by diluting 1 ml of concentrated ammonia (sp gr 0.90) to 100 ml with water.

4.3 Sodium Chloride — Solid.

4.4 Sulphuric Acid — 10 percent (*w/v*)

4.5 Sodium Carbonate — (a) 5 percent solution (*w/v*), and (b) solid.

4.6 Sodium Nitrate — Solid.

4.7 Sodium Hydroxide Solution — 1, 5, 10 and 30 percent (*w/v*).

4.8 Sodium Hydrosulphite — (a) 10 percent (*w/v*) freshly prepared , and solid (*see* IS 1919 : 1982).

4.9 Sodium Bisulphite — 30 percent solution (*w/v*).

4.10 Rectified Spirit — (*see* IS 323 : 2009).

4.11 Hydrogen Peroxide — 3 percent (*w/v*) (10 volumes).

4.12 Hydrochloric Acid — (a) prepared by diluting 15 ml of concentrated hydrochloric acid to 100 ml, and (b) concentrated.

4.13 Ethylenediamine — sp gr 0.97, b.p. 117 °C.

4.14 Ether

4.15 Bleached Cotton

4.16 Scoured Wool, Undyed

4.17 Dimethylformamide — (a) 50 percent (*w/v*), and (b) concentrated b. p. 152° to 154 °C.

4.18 Tannin Reagent — Prepared by dissolving 10 g of tannic acid and 10 g of anhydrous sodium acetate in 200 ml of water.

4.19 EDTA–Glycerine Solution — 4 percent (*w/v*) solution of disodium salt of ethylenediamine tetra acetic acid in glycerine.

4.20 Paraffin Wax

4.21 Blank Vat Solution — Prepared by dissolving 15 g of sodium hydroxide and 20 g of sodium hydrosulphite in sufficient water and made to 1 litre.

5 PROCEDURE

5.1 For preliminary identification of dyes on fibres, follow the procedure given in Annex A.

NOTE — While identifying the dyes used for dyeing of pale shades, it is advisable to use large specimen and larger quantities of reagents and to concentrate the extract before making the test.

ANNEX A

(*Clause* 5.1)

TESTS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON WOOL, SILK AND OTHER PROTIEN FIBRES

<table>
<tr><td colspan="8">The test specimen is successively treated with (a) 50 percent dimethylformamide, (b) concentrated dimethylformamide, and (c) a mixture of glacial acetic and rectified spirit (1 : 1, v/v) at boil for 3 to 4 minutes with intermediate washing with water and squeezing</td></tr>
<tr><td rowspan="6">No stripping or partial stripping of dye
REACTIVE DYES</td><td colspan="7">If dyes of group I are absent, treat the test specimen with 0.1 ml of glacial acetic acid and warm. Add 5 ml of water.</td></tr>
<tr><td rowspan="5">The acetic acid extract is distinctly coloured
BASIC DYES</td><td colspan="6">If dyes of groups I and II are absent, take a fresh test specimen and add to it 5 to 10 ml of 1 percent ammonium hydroxide solution. If extract is coloured, remove the stripped specimen and divide the extract into two portions. (a) Add to the first portion 30 mg of sodium chloride and 10 to 30 mg each of bleached cotton and scoured wool, and boil. (b) Neutralize the other portion by adding 10 percent sulphuric acid and few drops in excess. Add bleached cotton and scoured wool and boil.</td></tr>
<tr><td rowspan="4">Cotton is dyed in first portion
DIRECT DYES</td><td rowspan="4">Wool is dyed in second portion
ACID DYES <i>(If Direct and Basic Dyes absent)</i></td><td colspan="4">If dyes of groups I, II and III are absent, take a test specimen (about 5 g) and ash it completely in a porcelain crucible. Add 200 mg of flux (equal parts of sodium carbonate and sodium nitrate) and fuse.</td></tr>
<tr><td colspan="2">Test for the Presence of Metal
(Cr or Co present)
<i>Metal complex or Mordant dyes</i>
(<i>chrome</i>)
Cotton is dyed. In case of very pale dyeings it may be necessary to re-dye the cotton in 2 to 4 successive solutions stripped from the original dyeing
VAT DYES</td><td colspan="2">If dyes of groups I to IV are absent, take a test specimen (about 200 to 300 mg) and add 2.5 ml of 10 percent sodium hydroxide and boil until it is dissolved. Add 25 to 30 mg of sodium hydrosulphite, 20 to 50 mg of sodium chloride and 10 to 15 mg of bleached cotton. Keep near boil for 2 minutes and cool. Take out the cotton and place it on filter paper for 1 to 2 minutes and oxidize with sodium nitrate and acetic acid solution.</td></tr>
<tr><td colspan="2">Strip the dye in hot 1 percent ammonium hydroxide. After cooling acidify the solution with hydrochloric acid. Shake up the extract with ether</td><td rowspan="2">Cotton is undyed and colour is destroyed
AZOIC DYES</td><td rowspan="2">Cotton is undyed and colour is destroyed
AZOIC DYES</td></tr>
<tr><td>Ether is coloured
<i>Metal complex Dyes</i></td><td>Ether is not coloured
<i>Mordent Dyes (Chrome)</i></td></tr>
<tr><td>GROUP I</td><td>GROUP II</td><td colspan="2">GROUP III</td><td colspan="2">GROUP IV</td><td colspan="2">GROUP V</td></tr>
</table>

ADDITIONAL TESTS

a) *Basic Dye*

1) Take a test specimen. Add to it 1 ml of glacial acetic acid and warm. Add 5 ml of water. To the extract add tannin reagent. A coloured precipitate is obtained.

2) Take a test specimen. Boil it with rectified spirit; a coloured extract is obtained.

3) Take a test specimen, boil it in 2 ml glacial acetic acid, Add 30 percent sodium hydroxide till the solution is alkaline. A change in colour or complete decolourization is obtained. Acidify the solution with 5 percent acetic acid. The original colour is restored.

b) *Direct Dyes*

Take a test specimen and boil it with 5 percent sodium carbonate solution for half a minute in presence of few pieces of bleached cotton. The cotton is stained. The stained cotton is practically unaffected on treatment of 1 percent ammonium hydroxide solution at boil.

NOTES

1 In case of silk dyeings 5 to 10 percent sodium hydroxide should be used instead of sodium carbonate solution.

2 In the above test, certain dyes which are closely allied to substantive azo dyes in chemical structure, stain the cotton to a comparatively slight extent. Identification of such dyeings may be achieved by boiling the specimen in 1 percent sodium hydroxide solution when dull yellowish colour is obtained. This yellowish colour is also obtained on boiling the dyeings with 30 percent sodium bisulphite solution.

c) *Acid, Metal Complex and Mordant Dyes* (*Chrome*)

1) Take a specimen. Boil it with dimethylformamide. Bleeding indicates acid dyes, slight bleeding indicates metal complex dyes. No bleeding indicates mordant dyes (chrome).

2) Heat the test specimen in solution of EDTA in glycerine. At 140 °C : No change – *Acid and mordant dyes*. Rapid change (in 1 to 2 minutes) – 1 : 1 *Metal complex dyes* (acid dyeing). Slow change (in 20 min) – 1 : 2 *Metal complex dyes* (neutral dyeing). At 160 °C : No change – *Acid dyes*.

3) Take a specimen. Boil it with dilute hydrochloric acid. Take out the specimen, wash and treat it with 10 percent sodium hydrosulphite solution. The colour is destroyed.

4) Most of the after chrome dyes are not stripped. This fact should be taken as a clue for mordant dyes (chrome).

d) *Vat and Azoic Dyes*

1) Warm some paraffin wax in a white porcelain crucible until faint vapours appear. Take a test specimen and hold it in molten wax for about a minute. Remove the specimen. After cooling any staining of paraffin wax is readily seen against white background of the porcelain.

2) Take a test specimen and treat it with a blank vat solution at 60^0C in a test tube. Oxidize the specimen with hydrogen peroxide 3 percent (*m*/*v*).

   i) Change in colour and original colour restores on oxidation – *vat dyes*.

   ii) Test solution becomes yellow. Colour of the pattern usually remains unchanged. If the colour is changed and original colour not restored – *Azoic dyes*.

3) Take a test specimen. Warm it with ethylenediamine. Add aqueous solution of sodium hydrosulphite to ethylenediamine extract. The coloured extract is decolourized readily and permanently. – *Azoic dyes*.

NOTE — Most azoic dyes on wool and silk yield slimy residues of the same intense colours as the original dyes, on boiling dyes in 5 and 10 percent sodium hydroxide solution (distinction from mordant dyes). Most yellow dyeings and prints change to orange or red colours.

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

Organization	*Representative(s)*
Department for Jute and Fibre Technology, Institute of Jute Technology, University of Calcutta	Prof A. K. Samanta (***Chairman***)
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Ama Herbals, Lucknow	Shri Y. A. Shah
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Textiles Committee, Mumbai	Shri Kartikeya Dhanda Shrimati Shilpi Chauhan (*Alternate*)
The Arvind Mills Limited, Ahmedabad	Shri Rajarshi Ghosh Shri Umasankar Mahapatra (*Alternate*)
The Bombay Textile Research Association, Mumbai	Dr (Shrimati) Padma S. Vankar Shri M. P. Sathianarayanan (*Alternate*)
The South India Textile Research Association, Coimbatore	Dr Prakash Vasudevan Shri S. Sivakumar (*Alternate*)

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